



CERTIFICATION OF TRANSLATION

I, Yeon-sook Yun, an employee of Y.P. LEE, MOCK & PARTNERS of The Koryo Bldg., 1575-1 Scocho-dong, Seocho-gu, Seoul, Republic of Korea, hereby declare under penalty of perjury that I understand the Korean language and the English language; that I am fully capable of translating from Korean to English and vice versa; and that, to the best of my knowledge and belief, the statement in the English language in the attached translation of Korean Patent Application No. 10-2003-0011341 consisting of 17 pages, have the same meanings as the statements in the Korean language in the original document, a copy of which I have examined.

Signed this 17th day of February 2006

A handwritten signature in cursive script, reading "yeonsook yun", written over a horizontal line.

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ABSTRACT

[Abstract of the Disclosure]

5 Provided is a toner including a binder resin, a colorant, and an external additive
used in printers, fax machines, and copy machines, the toner including: 0.1 to 3.0 wt%
of large silica particles; 0.1 to 3.0 wt% of small silica particles; 0.1 to 2.0 wt% of
positively chargeable aluminum oxide particles having a charge amount of +50 to +500
μ C/g per a unit mass; 0.1 to 2.0 wt% of titanium dioxide microparticles; 80-95 wt% of a
binder resin; and 0.5 to 10 wt% of a colorant. Therefore, a thin toner layer with a
10 uniform toner amount is formed on a toner carrier to a thickness of 0.3 to 1.0 mg/cm² or
less and stable charge distribution and toner flowability are maintained for a long time,
resulting in prevention of fog and toner scattering, and improvements in developing
efficiency and toner durability.

15 [Indexed Words]

Toner, Silica, Titanium Dioxide, External Additive

SPECIFICATION

[Title of the Invention]

5 Toner for electrophotography

[Detailed Description of the Invention]

[Object of the Invention]

[Technical Field of the Invention and Related Art prior to the Invention]

10 The present invention relates to a toner used with electrophotography, and more particularly, to a toner used with electrophotography having improved developing efficiency in which two types of silica particles, a titanium dioxide (TiO_2), and a positively chargeable aluminum oxide (Al_2O_3) are used as external additives.

 As a developing method used in electrophotography, there are a two-component
15 developing method and a one-component developing method. The one-component developing method is subdivided into a magnetic one-component developing method and a non-magnetic one-component developing method. The magnetic one-component developing method is a method that uses a magnetic one-component developing toner. The non-magnetic one-component developing method is a method
20 that transfers a non-magnetic one-component developing toner layer formed on a developing roller to a photoconductor using a contact or non-contact developing method.

 Among various developing methods using a one-component developer, a contact-type, non-magnetic, one-component developing method has excellent cost
25 competitiveness, but provides poor dot reproducibility and line reproducibility, and low resolution, which renders creation of high quality images difficult. On the other hand, with respect to a non-contact-type, non-magnetic, one-component developing method, a simple developing apparatus is used, which promotes miniaturization of the apparatus.

Also, this method enables color reproducibility, edge reproducibility, good tone gradation, and high resolution printing, thereby providing high quality images.

A non-contact-type, non-magnetic developing method includes a charging operation, a light-exposing operation, a developing operation, a transferring operation, and a cleaning operation. In the case of a toner used in a conventional non-contact-type, non-magnetic developing method, a colorant, a charge control agent, and a releasing agent are uniformly dispersed in a binder resin to improve color, chargeability, and fixing properties. Also, various types of external additives are added to toner particles to impart functionality, such as flowability, charge stability, and cleaning properties, to the toner particles.

Japanese Patent Laid-Open Publication No. Hei.11-0095486 discloses a toner containing conductively treated silica particles as an external additive. Japanese Patent Laid-Open Publication No. Hei.11-295921 discloses a toner containing three types of silica particles with different particle sizes as an external additive.

In the non-contact-type, non-magnetic, one-component developing method, it is important to form a thin toner layer on a developing roller in a developing region, to prevent a fog (or background) in a non-image area and toner scattering. However, due to an increased charge amount of a toner during forming of a thin toner layer on a developing roller, developing efficiency is rapidly lowered, thereby decreasing an image density. Also, when the charge amount of a toner is adjusted to a low level to prevent the lowering of a developing efficiency, the increase of fog formation and contamination by toner scattering are likely to occur.

Therefore, stable maintenance of the charge amount and charge distribution of a toner as well as formation of a thin toner layer on a developing roller opposite to a developing area is required. This is accomplished by appropriately selecting the type and composition of external additives added to toner particles.

[Technical Goal of the Invention]

Accordingly, it is an aspect of the present invention to provide a toner with improved developing efficiency and durability, which is free from fog and toner scattering, by appropriately selecting the types and contents of external additives.

5 [Structure of the Invention]

According to an aspect of the present invention, there is provided a toner comprising a binder resin, a colorant, and an external additive used in printers, fax machines, and copy machines, the toner comprising as an external additive: large silica particles having an amount of 0.1 to 3.0 wt%; small silica particles having an amount of 10 0.1 to 3.0 wt%; positively chargeable aluminum oxide particles having an amount of 0.1 to 2.0 wt% having a charge amount of +50 to +500 μ C/g per a unit mass; titanium dioxide microparticles having an amount of 0.1 to 2.0 wt%; a binder resin having an amount of 80-95 wt%; and a colorant having an amount of 0.5 to 10 wt%.

The positively chargeable aluminum oxide particles have a charge amount of 15 +100 to +300 μ C/g.

The positively chargeable aluminum oxide particles have an average particle size of 0.1 to 3 μ m, and the titanium dioxide microparticles have an average particle size of 10 to 100 nm. In particular, the positively chargeable aluminum oxide particles can have an average particle size of 0.1 to 2.0 μ m, and the titanium dioxide microparticles 20 can have an average particle size of 15 to 80 nm.

The large silica particles have an average particle size of 20-200 nm, and the small silica particles have an average particle size of 5-20 nm.

The weight ratio of the large silica particles to the small silica particles is in a range of 1:1 to 3:1, for example, 1.5:1 to 2.5:1.

25 The binder resin has an acid number of 3 to 12 mgKOH/g.

Hereinafter, the present invention will be described in more detail.

The present invention relates to an additive including silicas having different particle sizes, positively chargeable aluminum oxide, and titanium dioxide.

In a toner of the present invention, large silica particles mainly act as spacer particles to prevent deterioration of the toner and improve transferability. Also, small silica particles mainly act to impart flowability to the toner. As the content of the large silica particles increases, M/A(mg/cm²) and flowability decrease. On the other hand, as the content of the small silica particles increases, M/A increases and fixing property decreases. The term, "M/A" as used herein indicates the weight of a toner per unit area measured on a developing roller after passing through a toner layer regulating member. Considering that improvement in fog prevention characteristics and toner scattering prevention can be accomplished at a low M/A, it is important to form a thin toner layer with M/A of 0.3 to 1.0 mg/cm². In this regard, improvement of toner properties can be accomplished by optimally adjusting the particle size, content, and combination ratio of the large silica particles and the small silica particles.

The large silica particles have an average particle size of 20 to 200 nm, preferably 30 to 150 nm.

If the particle size of the large silica particles is less than 20 nm, the large silica particles may be easily buried in a toner, which makes it difficult to act as spacer particles. On the other hand, if it exceeds 200 nm, the large silica particles may be easily separated from a toner and may not act as spacer particles.

The small silica particles have an average particle size of 5 to 20 nm, preferably 7 to 16 nm.

If the particle size of the small silica particles is less than 5 nm, the small silica particles may be easily buried in minute depressions of the surfaces of toner particles, and chargeability and flowability may not be easily controlled. On the other hand, if the particle size of the small silica particles exceeds 20 nm, flowability of a toner may be insufficient.

The large silica particles are used in an amount of 0.1 to 3.0 wt% relative to the weight of the toner particles. If the content of the large silica particles is less than 0.1 wt%, the large silica particles may not act as spacer particles. On the other hand, if the content of the large silica particles exceeds 3.0 wt%, the large silica particles may be

separated from a toner or may cause damage to the surface of a photoconductor, and an image resolution may be lowered.

The small silica particles are used in an amount of 0.1 to 3.0 wt% relative to the weight of the toner particles. If the content of the small silica particles is less than 0.1 wt%, flowability of a toner may be lowered. If the content of the small silica particles exceeds 3.0 wt%, the fixing property of a toner may be lowered and the charge amount of a toner may be excessively increased.

The combination ratio of the large silica particles to the small silica particles may vary according to a developing system. However, for the purpose of formation of a thin toner layer, the content of the large silica particles may be no less than that of the small silica particles.

In this regard, it is preferable to set the weight ratio of the large silica particles to the small silica particles at a range of 1:1 to 3:1, and more preferably 1.5:1 to 2.5:1.

When the weight ratio of the large silica particles to the small silica particles is greater than 1:1, M/A of 0.3 to 1.0 mg/cm² can be stably obtained, and fog and toner scattering can be prevented. If the content of the large silica particles is lower than that of the small silica particles, the thickness of a toner layer may increase(i.e., the weight ratio is less than 1:1), a charge amount may decrease, and a fixing property may be lowered. On the other hand, if the content of the large silica particles is excessively higher than that of the small silica particles(i.e., the weight ratio is more than 3:1), flowability of a toner may worsen.

To improve charge stability and flowability, a toner of the present invention includes aluminum oxide and titanium dioxide as an external additive

If inorganic titanium dioxide microparticles are used alone, the chargeability of a toner may be lowered during long-term usage, thereby causing toner scattering or uneven charge distribution. To solve these problems, positively chargeable titanium dioxide particles are added as an additional external additive.

The inorganic titanium dioxide microparticles impart flowability and uniform charge distribution to a toner, and the positively chargeable titanium dioxide particles

impart long-term charge stability to a toner. In this regard, the adjustment of the content and average particle size of these two components may be important, like the large and small silica particles. Appropriate selection of the resistance of these two components may also be important.

5 The positively chargeable aluminum oxide particles may have a charge amount of +50 to +500 μ C/g, preferably +100 to +300 μ C/g. If the charge amount of the positively chargeable aluminum oxide particles exceeds +500 μ C/g, uneven charge distribution of a toner and fog in a non-image area may occur. If it is less than +50 μ C/g, sufficient addition effect may not be obtained.

10 The positively chargeable aluminum oxide particles have an average particle size of 0.1 to 3.0 μ m, preferably 0.1 to 2.0 μ m. If the average particle size of the positively chargeable aluminum oxide particles is less than 0.1 μ m, chargeability of a toner may be lowered. On the other hand, if it exceeds 3.0 μ m, the positively chargeable aluminum oxide particles may be easily separated from a toner, and a developing
15 member such as a photoconductor may be easily damaged.

 The inorganic titanium dioxide microparticles have an average particle size of 10-100 nm, for example, 15-80 nm.

 When the average particle size of the inorganic titanium dioxide microparticles is less than 10 nm, chargeability decreases. On the other hand, when the average
20 particle size of the inorganic titanium dioxide microparticles is greater than 100 nm, flowability decreases.

 The inorganic titanium dioxide microparticles impart flowability to a toner, and the positively chargeable titanium dioxide particles impart long-term charge stability to a toner. In this regard, the adjustment of the content and average particle size of these
25 two components may be important, like the large and small silica particles. Appropriate selection of the resistance of these two components may also be important.

 The positively chargeable aluminum oxide particles are used in an amount of 0.1 to 2.0 wt% relative to the weight of the toner particles, and the inorganic titanium dioxide

microparticles are used in an amount of 0.1 to 2.0 wt% relative to the weight of the toner particles.

If the content of the positively chargeable aluminum oxide particles is less than 0.1 wt%, sufficient addition effect may not be obtained. On the other hand, if it exceeds 2.0 wt%, there may arise problems such as uneven charge distribution of a toner, poor fixing properties, an image fog due to separation from a toner, and damage to a developing member such as a photoconductor. When the amount of the inorganic titanium dioxide microparticles is less than 0.1 wt%, flowability decreases. On the other hand, the amount of the inorganic titanium dioxide microparticles is greater than 2.0 wt%, charge stability and fixing characteristics decrease.

Toner particles of the present invention include a binder resin.

Various known resins can be used as the binder resin. Examples of the binder resin include polystyrene, poly-p-chlorostyrene, poly- α -methylstyrene, styrene based copolymer such as styrene-chlorostyrene copolymer, styrene-propylene copolymer, styrene-vinyltoluene copolymer, styrene-vinylnaphthalene copolymer, styrene-methylacrylate copolymer, styrene-ethylacrylate copolymer, styrene-propylacrylate copolymer, styrene-butylacrylate copolymer, styrene-octylacrylate copolymer, styrene-methylmethacrylate copolymer, styrene-ethylmethacrylate copolymer, styrene-propylmethacrylate copolymer, styrene-butylmethacrylate copolymer, styrene- α -chloromethylmethacrylate copolymer, styrene-acrylonitrile copolymer, styrene-vinylmethylether copolymer, styrene-vinylethylether copolymer, styrene-vinylethylketone copolymer, styrene-butadiene copolymer, styrene-acrylonitrile-indene copolymer, styrene-maleic acid copolymer, and styrene-maleic ester, polymethylmethacrylate, polyethylmethacrylate, polybutylmethacrylate, and a copolymer thereof, polyvinylchloride, polyvinyl acetate, polyethylene, polypropylene, polyester, polyurethane, polyamide, epoxy resin, polyvinylbutyral resin, rosin, modified rosin, terpene resin, phenolic resin, aliphatic or cycloaliphatic hydrocarbon resin, aromatic

petroleum resin, chlorinated paraffin, and paraffin wax, and a mixture thereof. Among them, polyester resin has good fixing properties and is suitable for a color developer.

Preferably, the binder resin may be used in an amount of 80 to 95 wt% of the toner particles.

5 The properties of a toner according to the present invention are also affected by the acid number of the binder resin. As the acid number of the binder resin increases, adherence of a toner on a blade increases. In this regard, a low acid number is preferred. Preferably, the acid number of the binder resin is in a range of 3 to 12 mgKOH/g. If the acid number is less than 3 mgKOH/g, chargeability may be lowered.
10 On the other hand, if the acid number exceeds 12 mgKOH/g, stability of the charge amount of a toner with a change of humidity may be adversely affected, and adherence of a toner to a developing member may increase.

Toner particles of the present invention include a colorant.

Examples of the colorant include carbon black, aniline black, aniline blue,
15 charcoal blue, chromium yellow, ultramarine blue, dupone oil red, quinoline yellow, methylene blue chloride, phthalocyanine blue, malachite green oxalate, lamp black, Rose Bengal, rhodamine colorant or dye, anthraquinone dye, monoazo- and bisazo-dye, and quinachridone magenta dye. The colorant is used in a sufficient amount so that a visible image is formed to an appropriate density.

20 When carbon black is used as the colorant, it has preferably a primary particle size of 15 to 70nm, in particular 20 to 55nm, and specific surface area of 200 m²/g or less. Therefore, good dispersability and pulverizability of the colorant with other components during melting and/or blending are ensured.

 The colorant may be used in an amount of 0.5 to 10 wt%, preferably 0.5 to 8 wt%,
25 and more preferably 1 to 5 wt% of the toner particles.

If the content of the colorant is less than 0.5 wt%, a coloration effect may be insufficient. On the other hand, if it exceeds 10 wt%, even though an image density is saturated, the developing property of a toner may be lowered. For example, due to low

electric resistance of a toner, an insufficient amount of triboelectric charge may be obtained, thereby causing a fog.

Toner particles of the present invention may include a charge control agent (CCA) to enhance chargeability and a releasing agent such as wax to have better fixing property, which are uniformly dispersed in the binder resin.

It is required that a toner is stably adsorbed to the surface of a developing roller by an electrostatic force. Since the electrostatic force of a toner is generated by a charge blade, a fast charge speed is required. The charge control agent is thus necessary for the charge stability of a toner.

Examples of the charge control agent include useful dyes such as metal-containing azo dyes, salicylic acid metal complexes, nigrosin dyes, quaternary ammonium salts, triphenylmethane based control agents, and oil blacks, naphthenic acids, salicylic acids, octylic acids, and their metal salts such as manganese salts, cobalt salts, iron salts, zinc salts, aluminum salts, and lead salts, and alkylsalicylic acid metal chelates.

Preferably, the charge control agent is used in an amount of 0.1 to 10 wt%. If the content of the charge control agent is less than 0.1 wt%, sufficient addition effect may not be obtained. On the other hand, if the content of the charge control agent exceeds 10 wt%, charge instability may be caused.

Recently, low temperature fixing properties of a toner are required for low energy consumption and warm-up time reduction. For this, a releasing agent, such as wax that provides good fixing properties at a wide temperature range, is required.

The releasing agent (lubricant) may be a polyalkylene wax such as low molecular weight polypropylene and low molecular weight polyethylene, paraffin wax, high fatty acid, and fatty acid amide. The releasing agent is used in an amount of 0.1 to 10 wt%. If the content of the releasing agent is less than 0.1 wt%, sufficient addition effect may not be obtained. On the other hand, if it exceeds 10 wt%, there may arise problems such as poor offset resistance, low flowability, and caking.

The charge control agent and the releasing agent may be dispersed in toner particles or coated on toner particles. The former is general.

A toner of the present invention may further include a high fatty acid or a salt thereof to protect a photoconductor and to prevent deterioration of a developing property, thereby resulting in a high quality image.

Example

Composition of Toner Particles

Binder Resin

Polyester: 92 wt%

Acid Number: 7 mgKOH/g

Colorant (Carbon Black)

MA100 (Mitsubishi Chemical Co., Ltd.): 5 wt%

Charge Control Agent (Fe Complex)

Bontron S-34 (Orient Chemical Industries, Ltd.): 1 wt%

Releasing Agent (low molecular weight polypropylene wax)

660P (Sanyo Chemical Industries Ltd.): 2 wt%

Toner particles with the particle size of 8 μm were prepared using these above components according to a common toner preparation method and then added with following external additives to thereby obtain toner of the present invention:

Large Silica Particles (NAX50, Nippon Aerosol Co., Ltd.)

Average Particle Size 30 nm

Content 1 wt% based on 100 wt% of the untreated

toner

Small Silica Particles (H3005, Wacker-Chemie GmbH)

Average particle size 15 nm

Content 0.6 wt% based on 100 wt% of the untreated

toner

Positive Chargeable Aluminum Oxide (ET-500W, Ishihara Sangyo Kaisha, Co.)

Average Particle Size 500 nm

5 Content 1 wt% based on 100 wt% of the untreated

toner

Amount of Charge per unit mass +300 Ω cm

Inorganic Titanium Dioxide Microparticles (NKT90, Nippon Aerosol Co., Ltd.)

Average Particle Size 20 nm

10 Content 1 wt% based on 100 wt% of the untreated

toner

Comparative Example

15 Toner was prepared in the same manner as in Example 1 except that two types of silica particles and inorganic titanium dioxide microparticles were added to toner particles as external additives.

Experimental Example

20 Image qualities of the toners prepared according to Example and Comparative Example were evaluated using 20 ppm standard LBP printer. The image density (I/D), fog in a non-image area (B/G), and streak (vertical stripe type image fog due to adherence of the toner particles to a blade) of images were measured to evaluate the characteristics of the toner. Here, I/D was evaluated by measuring the density of a black pattern on a paper, B/G was evaluated by measuring the concentration of the toner on a non-image area of a photoconductor using a densitometer (SpectroEye, 25 GretagMacbeth Co.). Dot reproducibility and streak were evaluated by the naked eye.

The operational condition of a developing device was as follows:

Surface Potential (Vo): - 700 V

Latent Image Potential (VL): - 100 V

Developing Roller Applied Voltage

Vp-p = 1.8 KV, Frequency = 2.0 kHz,

Vdc = - 250 V, Duty Ratio = 40 % (Square Wave)

5 Developing Gap (GAP) : 250 μ

Developing Roller

(1) Aluminum

Roughness : Rz = 1 ~ 2.5 (after coated with nickel)

(2) Rubber Roller

10 Resistance: $5 \times 10^5 \Omega$

Hardness: 50

Toner

Charge Amount (q/m) = -14 to -20 μ C/g (measured on developing roller
after passing through a toner layer regulating member)

15 Amount of Toner = 0.4 to 0.8 mg/cm²

The above experimental results are presented in Tables 1 through 3 below.

The evaluation standard for I/D is as follows: "O": more than 1.3, " Δ ": 1.1 to 1.3, and
"X": less than 1.1.

20 The evaluation standard for B/G is as follows: "O": 0.14 or less, " Δ ": 0.15 to
0.16, and "X": 0.17 or more.

With respect to dot reproducibility and streak, "O" indicates good dot
reproducibility and little or no streak, " Δ " indicates acceptable dot reproducibility and
some streaks and "X" indicates poor dot reproducibility and many streaks.

25 Table 1
Evaluation Results of Toner of Example 1

N.P.* Section	Initial	2,000	4,000	6,000	8,000	10,000
I/D	O	O	O	O	O	O
B/G	O	O	O	O	O	△
Dot Reproducibility	O	O	O	O	△	△
Streak	O	O	O	O	O	O

* N.P.: Number of papers

Table 2

Evaluation Results of Toner of Comparative Example

N.P.* Section	Initial	2,000	4,000	6,000	8,000	10,000
I/D	O	O	O	O	△	X
B/G	O	O	O	△	X	X
Dot Reproducibility	O	O	O	△	△	X
Streak	O	O	O	△	△	X

* N.P.: Number of papers

As a result, it was found that inorganic titanium dioxide microparticles and positive chargeable aluminum oxide are used as an external additive, all of I/D, B/G, dot reproducibility, and streak characteristics were improved. In particular, as the number of papers increases, B/G and streak characteristics improved significantly.

[Effect of the Invention]

As seen from the above experimental results, when the two types of silica particles with different particle sizes, and the positively chargeable aluminum oxide and

titanium dioxide having different particle sizes were used as external additives, all of I/D, B/G, dot reproducibility, and streak characteristics were improved. In particular, as the number of printed papers increased, improvements in B/G and streak characteristics were excellent.

- 5 As apparent from the above descriptions, appropriate adjustment of the types and contents of toner external additives enables formation of a thin toner layer with a uniform toner amount (M/A) of 0.3 to 1.0 mg/cm² on a toner carrier. Therefore, stable charge distribution and toner flowability are maintained for a long time, thereby resulting in prevention of fog and toner scattering, and improvements in developing efficiency and
- 10 toner durability.

What is claimed is:

1. A toner comprising a binder resin, a colorant, and an external additive used in printers, fax machines, and copy machines, the toner comprising as an external additive:

5 large silica particles having an amount of 0.1 to 3.0 wt%;
small silica particles having an amount of 0.1 to 3.0 wt%;
positively chargeable aluminum oxide particles having an amount of 0.1 to 2.0 wt% having a charge amount of +50 to +500 μ C/g per a unit mass;
titanium dioxide microparticles having an amount of 0.1 to 2.0 wt%;
10 a binder resin having an amount of 80-95 wt%; and
a colorant having an amount of 0.5 to 10 wt%.

2. The toner of claim 1, wherein the positively chargeable aluminum oxide particles have a charge amount of +100 to +300 μ C/g.

15 3. The toner of claim 1, wherein the positively chargeable aluminum oxide particles have an average particle size of 0.1 to 3 μ m, and the titanium dioxide microparticles have an average particle size of 10 to 100 nm.

20 4. The toner of claim 1, wherein the positively chargeable aluminum oxide particles have an average particle size of 0.1 to 2.0 μ m, and the titanium dioxide microparticles have an average particle size of 15 to 80 nm.

25 5. The toner of claim 1, wherein the large silica particles have an average particle size of 20-200 nm, and the small silica particles have an average particle size of 5-20 nm.

6. The toner of claim 1, wherein a weight ratio of the large silica particles to the small silica particles is in a range of 1:1 to 3:1.

7. The toner of any one of claims 1 through 4, wherein a weight ratio of the large silica particles to the small silica particles is in a range of 1.5:1 to 2.5:1.

5 8. The toner of claim 1, wherein the binder resin has an acid number of 3 to 12 mgKOH/g.